Patented May 2, 1933

May 2, 1933

1,906,675

UNITED STATES PATENT OFFICE

WILLIAM E. WAGNER, OF ALTON, ILLINOIS, ASSIGNOR TO WESTERN CARTRIDGE COMPANY, OF ALTON, ILLINOIS, A CORPORATION OF DELAWARE

DOUBLE BASE PROPELLANT POWDER AND PROCESS OF MAKING THE SAME

No Drawing. Application filed April 7, 1930. Serial No. 442,472.

This invention relates to propellant powders of the double base type, such as nitro-cellulose-nitroglycerin powders. The illustrative embodiment hereafter described is particularly adapted for use as a rifle powder.

A progressive burning powder should have such characteristics as to cause a slow generation of gas during its initial stages of burning, in order to start the projectile charge into motion gradually; for a fast generation of gas before the projectile charge has moved any appreciable distance will result in high pressures due to the confined space in which the gas must be accommodated. As the projectile charge moves along the barrel and increases in velocity, the effective volume in the powder chamber is increased so rapidly that gas is not evolved at a rate sufficiently high to maintain a uniform pressure beyond the point at which the maximum pressure occurs. It is desirable that the initial pressure be limited, while the pressure along the barrel should be maintained. In order to maintain the pressure along the barrel, the core of the grain should be as fast burning as possible, while the outside surface of the grain should have its burning rate reduced. While the rate of ignition and, therefore, the initial rate of evolution of the gas can be controlled to a certain extent by coating or impregnating the powder grains, such as nitro-cellulose powder grains, with a deterrent such as dinitro-toluene, the bulk of the grain is of a uniform burning rate, so that the rate of the evolution of the gas does not increase sufficiently; accordingly, there is a too rapid fall of the pressure as the projectile charge moves along the barrel.

One of the objects of this invention, therefore, is to provide a double base powder grain which will meet the requirements noted above and overcome the limiting features of powder grains as heretofore produced.

Another object of this invention is to provide a double base powder grain, in which the rate at which the gas is evolved will progressively increase as the grain is consumed.

Further objects will appear from the detail description, in which there will be specifically described an illustrative embodiment of this invention. It is to be understood, however, that this invention is susceptible of various embodiments within the purview of those skilled in the art.

Generally stated, in accordance with this invention, a double base powder, such as nitro-cellulose-nitro-glycerin powder, is treated to partially extract one of the bases, such as nitro-glycerin, from the grain so that the percentage content of the partially extracted base and therefore the potential energy per unit weight of the powder will increase as the powder grains burn from the outside towards the center. This is accomplished in accordance with one embodiment of this invention, by treating the grain with an agent or solution which is a solvent for, say, the nitro-glycerin but which is a non-solvent for the larger part of the nitro-cellulose. While this results in a grain having generally the characteristic described, there will be left in the outer zone of the grain, even after extended treatment and upon evaporation of the solvent, part of the nitro-glycerin. In accordance with an embodiment of this invention the grain is treated with a deterrent or plasticizer. In order to accomplish still further the partial extraction of the nitro-glycerin from the surface of the grain and in order to secure the desired increase or taper effect from the surface to the interior of the grain, the double base grain is, after an initial treatment with a solvent for nitro-glycerin, which is a non-solvent for the bulk of the nitro-cellulose, treated in order to extract the dissolved nitro-glycerin in the solution remaining in and on the grain after the bulk of the solution has been removed. Thus after treatment to extract a portion of the nitro-glycerin in accordance with this invention the solution may have a nitro-glycerin content of from 6 to 12% and in the grain itself there will be a much more concentrated solution of nitro-glycerin. In order therefore to prevent this nitro-glycerin from being redeposited on and near the surface of the grains when the volatile solvent for nitro-

Oil, 149-140

glycerin left on the grain is evaporated, the powder is given an additional short wash of a fresh solution of nitro-glycerin solvent. The grain can then be dried to extract the solvent therefrom. While this treatment will result in a grain having the desired tapering effect, after an extended time under storage conditions, there may be a gradual migration of the nitro-glycerin which may tend to again distribute it throughout the grain. In accordance with an embodiment of this invention, therefore, the grain is treated with another gelatinizer to replace part of the nitro-glycerin which has been removed and which gelatinizer is preferably a deterrent and/or a plasticizer.

As an illustrative embodiment of this invention, we will take a double base powder containing 60% nitro-cellulose and 40% nitro-glycerin, produced in any manner well known to those skilled in the art. In a powder adapted more particularly for use in rifles of small calibre, the grains may be flat discs .050–.075 mm. thick and .90 mm. in diameter. These grains are allowed to soak in benzol, which is a solvent for nitro-glycerin but a non-solvent for nitro-cellulose. The soaking period is extended to the desired limit, five (5) hours being found practical. At this point the grain can be treated with a suitable gelatinizer which is preferably a deterrent and plasticizer, such as dibutyl phthalate in a suitable solvent such as benzol. The grain can then be dried. The time of soaking is dependent on the nature of the solvent and the desired effect, for example, when methanol is used, the time of extraction may be as low as five minutes.

In order to attain more fully the objects of this invention, the grains are soaked for a short period in a fresh solution of benzol in order to more completely extract the nitro-glycerin; this is preferably accomplished before the grains are dried and immediately after they have received their first treatment with benzol. The time of treatment will, of course, vary with the character of solvent used; thus in the case of benzol the treatment can be carried out for about five (5) minutes. The grains can then be dried in any suitable manner so as to evaporate the solvent, but preferably in a closed system with a solvent recovery. The grains can then be treated with a suitable gelatinizer, preferably a deterrent and plasticizer such as dibutyl phthalate. For this purpose to 100 parts by weight of powder treated for the removal of nitro-glycerin are added 70 parts by weight of a 6.75% solution of diamyl phthalate in benzol preferably in a rotating barrel with solvent recovery system. The benzol is driven off leaving the diamyl phthalate in the grain in combination with the nitro-cellulose and the remaining nitro-glycerin. After the grain is dry it can be graphited in the usual manner.

It has been found that a double base powder treated as above described will attain the objects of this invention. The progressive burning quality imparted to the powder allows the powder charge of the cartridges to be increased without increasing the maximum pressure, resulting in much higher velocities without any increase in pressure. This is shown by the following tests performed with the powder as heretofore described in comparison with the untreated powder, the test being conducted with a .22 long rifle cartridge; the results being the averages of forty shots.

Powder	Charge (grams)	Average pressure	Average velocity
Untreated	.80	13,704	892
Treated	2.35	13,708	1,231

A further test showed that the high velocity bullet with treated powder penetrated twice as far into wood as the lower velocity bullet fired with the untreated powder. A microscopic examination of the untreated double base grain reveals the fact that the interior of the grain does not consist of a uniformly gelatinized mixture of nitro-glycerin and nitro-cellulose but suggests that there are centers scattered through the body of the grain which are very high in nitro-glycerin content with inter spaces correspondingly low.

A microscopic examination after the initial treatment with benzol to remove nitro-glycerin indicates that the benzol attacks these high centers with great difficulty which accounts for the observed fact that extended treatment does not have much effect in removing more than about 60% of the original content of nitro-glycerin and also explains why grains many times thicker can be successfully treated and enough nitro-glycerin removed for the successful application of this invention in the same period of time as for the thinner grains. The extraction of nitro-glycerin decreases with the penetration and is less, adjacent to the centers of high nitro-glycerin content, in accordance with the well known scientific fact that the rate of diffusion of two liquids depends upon the difference of concentration; accordingly the more nitro-glycerin the solvent contains at any point within the grain, the less active it becomes in removing nitro-glycerin. Accordingly the undissolved nitro-glycerin left in the grain will taper from the outside towards the center with centers of high nitro-glycerin content distributed throughout the mass, and separated from each other by nitro-cellulose from which the nitro-glycerin has been largely removed. The depth of penetration and the extent to

which the nitro-glycerin is removed from the body of the grain will depend upon the character of the solvent and the period of treatment.

A microscopic examination of the grain after the final treatment with benzol indicates that the plasticizer distributes itself throughout the entire mass of the grain, being more concentrated in the areas of low nitro-glycerin content and less concentrated where more nitro-glycerin has remained in the grain. It is seen, therefore, that the resulting powder grain has an exterior of a slow burning combination of nitro-cellulose with a deterrent which is also a gelatinizer and plasticizer, and that from the outside towards the center of the grain and also toward the centers of high nitro-glycerin content there is a gradual decrease of deterrent and increase of nitro-glycerin which results in a progressive burning powder characterized by the fact that the rate of gas evolution increases as the grains burn from the outside towards the center.

While this invention is particularly applicable to nitro-cellulose-nitro-glycerin powders, gelatinizers for nitro-cellulose, other than nitro-glycerin, may be employed, such as nitrates of polyglycerin, or any of the glycols or polyglycols, or nitrates of any of the aromatic series or in fact any substance which when mixed with or gelatinized with nitro-cellulose increases the rate at which gas is evolved over powder gelatinized by the customary solvents, such as acetone or ether-alcohol. It will be understood, of course, that the nitro-cellulose may be of varying degrees of nitration best suited for the particular substance with which it is combined and the purpose for which it is employed. The solvent employed may be benzol, ethyl alcohol, methyl alcohol, ether or any other suitable solvent or mixture of solvents adapted for that purpose. The solvent may be one which may slightly dissolve the nitro-cellulose but not sufficiently to cause the grains to stick together. The gelatinizer may be dibutyl phthalate, diamyl phthalate, tricresyl phosphate, butyl stearate, dinitro-toluene, dimethyl diphenylurea or any other suitable deterrent or plasticizer.

The deterrent or plasticizer may be applied with any suitable solvent which may be a solvent only for the plasticizer without being a solvent for nitro-glycerin or nitro-cellulose such as carbon tetrachloride in the case of diamyl phthalate or it may be a solvent for the plasticizer and the nitro-glycerin as in the case of benzol, or it may be a solvent for the plasticizer, nitro-glycerin and a solvent for lower grades of nitro-cellulose, such as methyl alcohol, or a mixture of a solvent and non-solvent for nitro-cellulose, such as ethyl acetate and benzol in such proportions that it will not actively attack the nitro-cellulose, causing the powder grains to adhere one to the other.

It will furthermore be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and sub-combinations; that is contemplated by and is within the scope of the appended claims. It is, furthermore, to be understood that various changes may be made in details and operations within the scope of the appended claims, without departing from the spirit of this invention. It is, therefore, to be understood that this invention is not to be limited to the specific details and operations described.

Having thus described the invention, what is claimed is:

1. A process of treating double base powders, comprising, partially extracting the more active base from a gelatinized part of a double base grain.

2. A process of treating double base powders, comprising, partially extracting the more active base from the exterior of a double base grain.

3. The process of controlling the ignition or burning characteristics of a double base powder grain by partial extraction of the more active base from the exterior of the grain.

4. A process of treating double base powders, comprising, partially extracting the more active base from the grain and replacing the extracted base with a gelatinizer for the other base.

5. A process of treating double base powders, comprising, partially extracting the more active base from the grain and replacing the extracted base by a slower-burning component.

6. A process of treating double base powders, comprising, partially extracting the more active base from a double base grain so that the content of the partially extracted base will decrease from the interior to the surface of the grain.

7. A process of treating double base powders, comprising, partially extracting the more active base from a double base grain so that the content of the partially extracted base will decrease from the interior to the surface of the grain, and treating the grain with a deterrent.

8. A process of improving the ballistic properties of double base powders, comprising, removing part of the more active base by treating a double base grain with an agent which is a solvent for the more active of the bases but a substantially non-solvent for the other base.

9. A process of treating nitro-cellulose-nitro-glycerin powder grains, comprising, partially extracting the nitro-glycerin from the grain.

10. A process of treating nitro-cellulose-

nitro-glycerin powder grains, comprising, partially extracting the nitro-glycerin from the grain and replacing the extracted nitro-glycerin with a slower-burning component.

11. A process of treating nitro-cellulose-nitro-glycerin powder grains, comprising, partially extracting the nitro-glycerin from the grain so that the nitro-glycerin content will decrease from the interior to the surface of the grain.

12. A process of improving the ballistic properties of nitro-cellulose-nitro-glycerin powder grains comprising, removing part of the nitro-glycerin by treating the grain with a solvent for nitro-glycerin which is substantially non-solvent for nitro-cellulose.

13. A process of treating nitro-cellulose-nitro-glycerin powder grains, comprising, removing part of the nitro-glycerin by treating the grain with a solvent for nitro-glycerin which is substantially non-solvent for nitro-cellulose and treating the grain to extract the dissolved nitro-glycerin therefrom.

14. A process of treating nitro-cellulose-nitro-glycerin powder grains, comprising, treating the grain with a solvent for nitro-glycerin substantially non-solvent for nitro-cellulose and treating the grain with a deterrent.

15. A double base propellant powder grain the content of the more active base of which decreases from the interior to the surface of the grain.

16. A double base propellant powder grain the content of the more active base of which decreases from the interior to the surface of the grain and which is impregnated or coated with a slower-burning component.

17. A nitro-cellulose-nitro-glycerin powder grain whose nitro-glycerin content is reduced near the surface of the grain.

18. A nitro-cellulose-nitro-glycerin powder grain whose nitro-glycerin content is reduced near the surface of the grain and which is impregnated or coated with a deterrent.

19. A process of making double base powders, comprising, partially extracting the more active base from the grain and replacing the extracted portion with an ingredient having a different combustion rate than the extracted base.

In testimony whereof I affix my signature this 1st day of April, 1930.

WILLIAM E. WAGNER.

CERTIFICATE OF CORRECTION.

Patent No. 1,906,675.

May 2, 1933.

WILLIAM E. WAGNER.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, lines 109 and 110, for "successively" read "successfully"; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 1st day of August, A. D. 1933.

(Seal)

M. J. Moore.

Acting Commissioner of Patents.